Ag decorated SiO_2 Microspheres: Synthesis and their Antimicrobial activity

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Abstract-Silver (Ag) decorated on the surface of SiO_2 microsphere was achieved in presence of hydrazine hydrate by the chemical method. The synthesized Ag-SiO_2 was studied by various techniques like Ultraviolet-visible spectrophotometer, X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and Transmission Electron Microscopy (TEM). The broad peak at the wavelength of~425 nm confirmed the formation of Ag on to the SiO_2 surfaces. Hybrid nanostructure of Ag-SiO_2 was analyzed using field emission scanning electron microscopy which shows the Ag nanoparticle decorated on the surface of the SiO_2 nanoparticles and their diameter is in the microscale. Hybrid nanostructure of Ag-SiO_2 could be suitable material for the medical applications such as antiviral and antibacterial activity.

Keyword—Ag nanoparticles, SiO_2, Hybrid nanostructure, Reduction.

I. INTRODUCTION

Nanomaterials have increasing interest within the researchers and have shown extensive attention to the field of technological importance and scientific applications [1,2]. Silver compounds have been exploited for their medicinal properties for centuries and it has been known for long time that silver is an effective antimicrobial agent [3]. Silver sulfadiazine is used for topical treatment of burn-wounds and silver nitrate is still used as a prophylaxis in neonatal ophthalmic. Silver nanoparticles have been used as a catalyst for reduction of aromatic compounds [4]. Mono and bimetallic particles in the nanosize regime find extensive applications in catalysis, since with reduced size, increased surface area increases catalytic activity [5]. SiO2 have proven to be the most suitable for widespread environment application for its high chemical stability, non-toxicity, low cost and excellent degradation of organic pollutants [6]. The silica shells not only enhance the colloidal stability but also control the distance between core particles within assemblies through shell thickness.

Ag nanoparticles is synthesized by using various methods such as chemical method using sodium borohydride, alcohol at elevated temperature, γ-irradiation, push-pull method and photochemical method, thermal decomposition method and radiolytic methods [7]. The antimicrobial activity of Ag nanoparticles is the result of the interaction of silver ions with the three main components of the bacterial cell: the peptidoglycan cell wall and plasma membrane, bacterial DNA and bacterial protein, particularly enzymes [8]. Silver ions have long been known to have strong inhibitory and bactericidal effects, as well as excellent antibacterial performance towards bacteria such as Escherichia coli and Pseudomonas aeruginosa and Staphylococcus aureus [9].

In this paper Ag-SiO_2 nanocomposite were prepared by chemical method using hydrazine hydrate. Ag-SiO_2 was characterized by UV-Vis spectroscopy, X-Ray Diffraction (XRD), Field Emission Scanning Microscope (FESEM) and Transmission Electron Microscope(TEM). Furthermore, we have utilized the nanocomposite to test the anti-bacterial activity.

II. EXPERIMENTAL

Silicon tetrachloride ($SiCl_4$) (Alfa Aesar), N,N-Dimethylformamide (DMF) (Vetec), Silver nitrate($AgNO_3$) (Merk), Ammonium hydroxide (NH_4OH) (Rankem), Hydrazine Hydrate($N_2H_4.H_2O$) (CDH) and Ethanol was purchased as Analytical grade.

Synthesis of Ag decorated Silica microparticles

Silica microparticles were synthesized by $SiCl_4$ reacting with hydrazine hydrate along with N,N-dimethyl formamide (DMF). 1 ml of $SiCl_4$ was added to 20 ml of DMF containing 2.5 ml of hydrazine hydrate stirred at room temperature for 1 hour. The obtained white colloidal solutions were centrifuged and washed with deionized water and acetone.

2.5 ml of 0.1 M $AgNO_3$ were dissolved in 30 ml of deionized water with few drops of NH_4OH for adjust the pH ~ 10. The $AgNO_3$were slowly added with the obtained SiO_2 and stirred at 70°C for 30 min. The resulting Ag decorated SiO_2 colloidal solutions were centrifuged and washed with water and dried at 100°C

for 1 hour and redispersed in ethanol for further analysis.

Instrument

UV-Vis absorption spectrum was measured on Perkin-Elmer 650 spectrophotometer. XRD analysis were performed on Rich Siefert 3000 diffractometer with Cu-K(alpha)1 irradiation (=1.5406°A). The morphology and size of the samples were analyzed by FESEM using a HITACHI SU6600 field emission-scanning electron microscopy and TEM observations were performed on a HITACHI, H-7650 (Japan) instrument operated at an accelerating voltage of 100kV.

III. RESULT AND DISCUSSION

Synthetic Method for Ag decoration on the Surface of SiO_2 Microparticles

Alkaline condition produces strong nucleophiles that deprotonate hydroxyl ligands (-OH), and then these nucleophilic parts react with electrophilic materials such as Ag, Cu, Au, etc. Ag-SiO_2 nanocomposites are synthesized by three steps. The first step is deprotonation of hydroxyl ligand in SiOH. Addition of hydrazine into SiOH generates the nucleophilic SiO^- and the surface covered with base. The second step is electrophilic attack by the Electrophilic metal (Ag^+), which is easily bonded with the nucleophilic part (SiO^-). The third step is growth of the Ag nanoparticles by attaching more Ag^+ on the surface of the SiO_2 nanoparticle. The Ag forming process is considered to be sensitive to the configuration of terminal groups on the SiO_2 surface, since such surface groups (hydrazine) obviously provide the capability required for the reduction of Ag ions. Terminal OH groups usually form on the oxide surface by dissociative adsorption of water molecules, depending on their coordination symmetry. Accordingly, nanocomposite may form by auto reduction of noble metal ions with oxide surfaces. The efficiency of the surface-mediated reduction process can be decreased with consumption of hydroxyl groups and generation of surface charges [10].

UV-Vis absorption spectrum of Ag-SiO_2 nanoparticles were shown in Fig.1. UV-Vis Spectrum of Ag-SiO_2 nanoparticles showed absorption band at 425 nm. The silica colloids did not show any UV-Visible absorption, but the composite material show absorption peak at around 425 nm due to the Mie plasmon resonance from the silver nanoparticles.

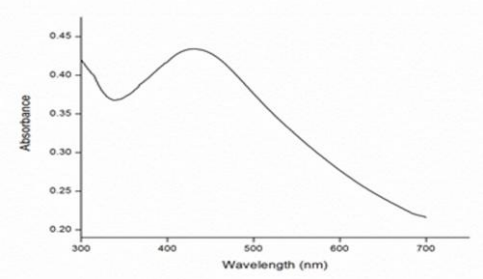

Fig.1. UV-Vis spectrum of Ag-SiO_2.

The position and shape of the plasmon absorption of silver nanoparticles are strongly dependent on the particle size, dielectric medium and surface adsorbed species. This is concerned with red-shifted absorption band of Ag-SiO_2 with reported results [11].

The X-Ray diffraction pattern showed in Fig.2 corresponds to the Ag-SiO_2. XRD pattern showed the weak diffraction peaks at 38° (111), 44° (200) and 64° (220). The peaks were compared with online database JCPDS-89-3722 was exactly match with XRD pattern which have face centered cubic (FCC) structure. The X-Ray diffraction pattern obtained for Ag-$SiO_2$was slightly amorphous in nature with broad peaks which confirms the Ag particles on the surface of SiO_2could be smaller in size.

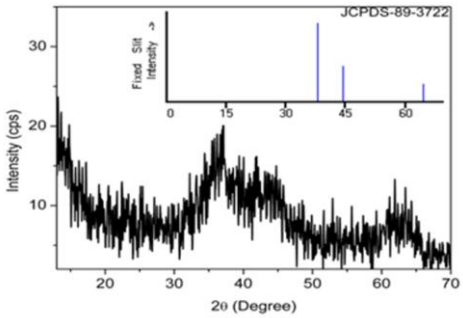

Fig.2. XRD pattern of Ag-SiO_2.

The FESEM of Ag-SiO_2 was shown in Fig.3. The SiO_2 was spherical with micro scale. Ag nanoparticles decorated on the surface of the SiO_2 microspheres were found by the bright spots spreads on the surface of SiO_2. The obtained results well matched with the TEM images of Ag-SiO_2 nanoparticles were shown in Fig.4. Ag-SiO_2 particle size was about ~2 µm. TEM image shows Ag nanoparticles were homogeneously spread on SiO_2 microspheres. TEM image also shows the SiO_2 was spherical in shape and the black dots corresponds to Ag nanoparticles.The size of the SiO_2 spheres were approximately 2 µm and the size of Ag dark spots decorated on the surface were approxiamtely 32 nm. From the FESEM and TEM images, one can conclude that the Ag nanopatiles uniformly decorated on the surface with nanoscale.

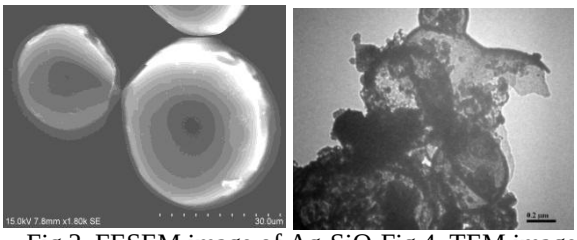

Fig.3. FESEM image of Ag-SiO_2Fig.4. TEM image of Ag-SiO_2

Antibacterial Activity

Antibacterial activity was demonstrated using a modification of the method originally described by Bauer et al. (1966) which is widely used for the

antibacterial susceptibility testing [12]. A loop full bacterium was taken from the stock culture and dissolved in 1 ml of Lb broth kept for incubation 12 hrs. Ag-SiO_2 was tested for antibacterial activity by agar well-diffusion method against Pseudomonas aeruginosa, Escherichia coli (Gram-negative bacteria), Bacillus subtilis and Staphylococcus aureus, (Gram-positive bacteria). The pure cultures of bacteria were swabbed uniformly on the individual plates using sterile cotton swabs on the Muller Hinton Agar. Six wells were made on 6 mm in diameter in Muller Hinton agar plates with help of gel puncture, different concentration like 25 µg, 50 µg, 75 µg, 100 µg, blank as DMSO and 10 µg of antibiotic (Streptomycin) were added in respective wells. Streptomycin was used as a positive control. The plates were incubated at 37°C for 24 hrs to observe formation of zone of inhibition.

Ag-SiO_2 was tested with Pseudomonas aeruginosa, Escherichia coli (Gram-negative bacteria), Bacillus subtilis and Staphylococcus aureus, (Gram-positive bacteria). The Pseudomonas aeruginosa shows a very good inhibition than commercial Streptomycin shown in Fig.5.(d) and also the diameter in the Table.1 Escherichia coli is good inhibition and Staphylococcus aureus and Bacillus subtilis is less inhibition.

Table 1. Inhibition zone diameters of Ag-SiO_2

Micro Organism	Streptomycin 10 µg	25 µg	50 µg	75 µg	100 µg
Staphylococcus aureus,	16mm	9mm	9mm	10mm	14 mm
Bacillus subtilis	8mm	6mm	6mm	4mm	4mm
Escherichia coli	12mm	13 mm	15mm	16mm	17 mm
Pseudomonas aeriginosa	10mm	10 mm	18mm	20mm	21 mm

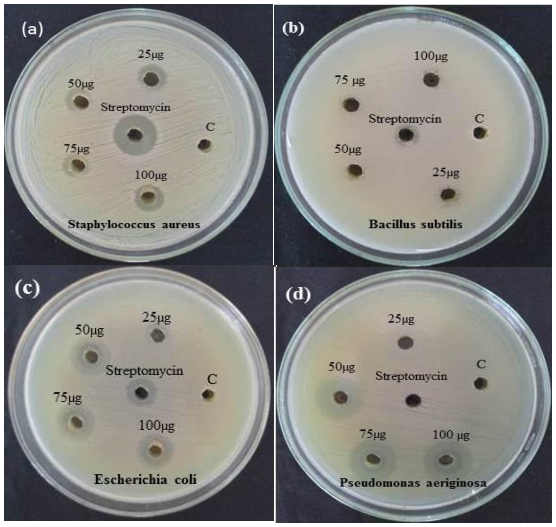

Fig. 5. Antibacterial activity of Ag-SiO_2

IV. CONCLUSION

Ag nanoparticles decorated on the surface of SiO_2 was prepared by chemical method at optimum temperature. The Ag-SiO_2 composite shows the absorption peak at around 425 nm due to the Mie plasmon resonance of the silver nanoparticles. XRD pattern shows the peaks at 38° (111), 44° (200) and 64° (220). The peaks were compared with online database JCPDS-89-3722 was exactly match with decorated on the surface of SiO_2. Antibacterial activity of Ag-SiO_2 were tested with micro-organisms, Pseudomonas aeruginosa shows a very good inhibition than the commercial streptomycin.

V. ACKNOWLEDGEMENT

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VI. REFERENCES

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